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PATENT APPLICATION

HILL & SCHUMACHER

Title: PLASTIC WOOD FIBER FOAM STRUCTURE AND
METHOD OF PRODUCING SAME

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PLASTIC WOOD FIBER FOAM STRUCTURE AND

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CROSS REFERENCE TO RELATED PATENT APPLICATION

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This patent application relates to United States Provisional Patent Application Serial No. 60/288,445 filed on May 4, 2001 entitled PLASTIC WOOD FIBER FOAM STRUCTURE AND METHOD OF PRODUCING SAME which is

10   incorporated herein by reference.

FIELD OF INVENTION

This invention relates to the continuous production of plastic/wood fiber composite foams, in an extrusion process, and is more particularly directed to

15   increasing the number of cells and reduction of cell size and thereby, improving the distribution of cells in the plastic matrix.

BACKGROUND OF THE INVENTION

The end of twentieth century is characterized by a large increase of

20   thermoplastic consumption with the consequent increase in their prices [1,2]. This situation has created an impetus for cost reduction by utilizing fillers in thermoplastic to form composite materials[3]. Composite materials also offer the opportunity to utilize the favorable properties of the constituent components [1-3].

The cost of wood-fibers is substantially lower than the other commonly used fibrous filler materials such as glass, mica and the like. Plastic/wood-fiber composites exhibit improved stiffness in addition to their lower costs. They can be a cost effective alternative to many filled plastics or metals in terms of bending stiffness or weight [3].

5   The wood-fibers are non-abrasive so that relatively large concentrations can be incorporated into plastics without causing serious machine wear during blending and processing.

Although plastic/wood-fiber composites have been commercialized, and can compete with certain plastic and wood products, their potential industrial applications

10   have been limited because of their low impact strength and high density compared to natural wood and certain plastics [4].

Impact strength and ductility of plastics can be significantly improved by incorporating a fine cell structure into them [5]. Additional benefits of having a foamed structure are the reduction in weight and cost. Therefore, it is reasonable to expect

15,   that, if a fine-celled structure is successfully produced in plastic/wood-fiber composites, the problems with impact strength and weight mentioned earlier can be reduced or eliminated. This would significantly increase the potential applications of these composites.

Water vapor adsorbed in the wood fiber is released during heating (plasticizing)

20   stage of extrusion, and is retained in the melt in a gaseous or liquid state until the extrudate comes out of the die. This results in the foam structure being very non-uniform. To get a good foam structure, it is preferable to maximize the extraction

(purging out) of the moisture from the wood fiber before the final processing of the

composite foam in the extruder. However, even oven-dried wood-fiber releases

additional moisture or other volatile gases when it is further heated to the processing

temperature of the plastics, which is typically above 180°C. This has been shown in a

5   thermo gravimetric analysis (TGA) study [22]. The fibers were initially heated at 110°C

for about 150 minutes so that they were completely dry. At this point, the temperature

was raised to 205°C, and it was observed that the wood-fibers lost an additional 3%

weight. Therefore it is not sufficient to define the moisture content in terms of the

conventional moisture content formula used for lumber wood. This additional moisture,

10   or volatile gases should be taken into account for foaming applications.

A thorough review of the extrusion process is provided by Rauwendaal [6]. The

processing of wood-fiber composites is a well-known art, and is described in many

patents.

There are a number of patents [7-9] directed to producing wood fiber/plastic

15   composite pellets in which the fibers are "encapsulated" by the plastic. The

encapsulation is achieved by processing the mixture at a temperature above the

melting point of the plastic. The lowest temperature at which thorough "wetting" occurs

is designated as the "encapsulation point". These pellets are then processed in

extruder to make the finished product profile. However, the major draw back with it is

20   that it is batch processing and would involve additional costs associated with such

processes.

Turk et al [10], describes a process for preparing a natural fiber and

thermoplastic composite, in which they used a vented extruder to remove the gaseous products and moisture released by the fibers. They describe a number of modifications carried out to prevent the loss of the extrudate from the vent. Even after all the modifications, an "interfacial agent" was required for overcoming this problem. This

5   suggests that different materials with different compositions may require different "interfacial agent". Therefore, for many compositions suitable "interfacial agent" may not exist.

Deaner et al [11-14] first produced pellets of plastic wood-fiber composites, which were subsequently used for profile extrusion. They describe the importance of

10   reducing the moisture content from the wood-fiber at the pelletizing stage. The moisture in wood fiber is reduced to less than 8 wt-% at this stage. At the final extrusion stage, the moisture is again removed using a vent under 3 inches of vacuum. The final product produced using this process is, however, heavier than the wood it replaces.

15   Pelikan et al [15] describe a process of producing cellular cavities in which the bubble-forming agent is bound to a carrier, such as vegetable or wood fibers, by molecular or capillary action. In this case the blowing agent is water, it is released at a temperature higher than 120 C, and is thus released more gradually leading to a uniform distribution of closed cells within the plastic matrix.

20   Boutillier [16] and Cope [17,18] describe production of plastic/wood-fiber composite foamed profiles using the so-called Celuka process. The outer surface of the extrudate is cooled, below the softening temperature of extrudate, upon exiting

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from the die. The solidified skin prevents the expansion in the outward direction and the material expands inwards, into a hollow cavity created by a solid body inside the die. However, it is difficult to control the expansion process and the foam structure obtained is non-uniform.

5   Accordingly, there is clearly a need for foamed thermoplastic/wood-fiber composites, which exhibit fine-celled structure and have easy processability. As mentioned earlier, the removal of moisture from the wood-fiber is necessary to obtain controlled expansion and uniform distribution of the cell structure throughout the product. Further, it should be removed at the highest processing temperature or as

10   close to this temperature as possible. One of the fundamental problems is that the moisture and volatiles from even the dried wood-fibers are typically gradually released, as long as it is being heated. No matter how much it has been devolatilized, these volatiles typically contribute to the foaming of plastic/wood-fiber composites. Accordingly, it would be advantageous to minimize the effect of the moisture and

15   temperature release by minimizing the H_2O component at the foaming stage.

SUMMARY OF INVENTION

In one aspect of the invention a process for producing plastic/wood fiber composite foamed structures includes the steps of pre-drying wood fiber filler; mixing it

20   with plastic to form a mixture; feeding the mixture into an extruder; introducing and mixing a blowing agent; subject the mixture to high shear forces and extruding the mixture to produce a plastic/wood fiber composite foamed structure. The wood fiber

filler has a degradation temperature and an active volatization temperature. During the pre-drying step the temperature is maintained below the degradation temperature to produce dried wood fiber filler. During the mixing step the dried wood fiber filler the mixing temperature is maintained below an active volatilizing temperature. During the

5 introducing and mixing step a blowing agent is introduced into the plastic/wood fiber mixture and is mixed therewith to produce a plastic/wood fiber/gas mixture. During the subjecting step the plastic/wood fiber/gas mixture is subjected to high shear forces in the presence of high pressures and the processing temperature is maintained below an active volatilizing temperature.

10 In another aspect of the invention a process for producing plastic/wood fiber composite foamed structures includes the steps of pre-drying wood fiber filler; mixing it with plastic to forma a mixture; feeding the mixture into and extruder; mixing a physical blowing agent with the mixture; subjecting the mixture to high shear forces and extruding the mixture. In the mixing step the dried wood fiber filler is mixed with plastic

15 to produce a plastic/wood fiber mixture. In the feeding step the plastic/wood fiber mixture is fed into an extruder. In the mixing step a physical blowing agent is mixed into the plastic/wood fiber mixture to produce a plastic/wood fiber/gas mixture. In the subjecting step the plastic/wood fiber/gas mixture is subjected to high shear forces in the presence of high pressures. Thereafter the plastic/wood fiber/gas mixture is

20 extruded to produce a plastic/wood fiber composite foamed structure.

The present invention encompasses the use of both, chemical blowing agents (CBA) and physical blowing agents (PBA), for producing fine-celled plastic/wood-fiber

composite foams. Uniformly distributed fine cells enhance material properties such as tensile strength, toughness, impact strength, insulation and surface quality, in addition to reduction in cost.

The present invention utilizes a CBA or a PBA to generate gaseous material,

5   which is dispersed or dissolved in the polymer matrix under high temperature and pressure. The dispersed gases become nuclei for the bubbles when the pressure is rapidly reduced at the die exit, whereas, the dissolved gases produce a large number of nuclei when subjected to a rapid pressure drop and/or sheer stress, leading to Spinodal decomposition of the dissolved gases from the polymer matrix [19]. But

10   these effects result in the creation of large number of bubbles which leads to the production a very fine cell structure in plastic/wood-fiber composite foams. The cell size and uniformity of distribution achieved with this invention, for producing wood-fiber/plastic composite foams, is far superior to what has been obtained in the prior arts.

15   A fine-celled structure is produced, by first dissolving the foaming gases into the polymer matrix, creating a large number of nucleation sites by using a nucleating agent and/or dispersed gas bubbles and/or a rapid pressure drop and/or a shear stress at the die exit and controlling the expansion by controlling the temperature of the extrudate melt and surface. If any gaseous material is not dissolved in the polymer matrix, it

20   remains present in the form of liquefied bubbles within the polymer matrix and the resultant foam structure exhibits these bubbles, with enlarged size, superimposed upon the uniformly distributed small bubbles. This results in a composite with relatively

a large cell size and a non-uniform cell structure.

As mentioned earlier, moisture and other volatile gases are released from the wood-fiber, even after it has been dried and/or devolatilized, whenever its temperature is elevated beyond or around the drying temperature. The solubility of H_2O is very low

5   in polymer and so, if it is present, it may remain un-dissolved in the melt and deteriorate the cell morphology. Also, the volatile gases may cause formation of big voids in the final products if the processing pressure is not high enough compared to the solubility pressures of the volatile gases. Accordingly, by determining the critical drying and processing temperature, the H_2O and volatile emissions are minimized as

10   much as possible to achieve this goal becomes crucial.

In order to determine a suitable drying and processing temperatures, the gravimetric behavior of wood-fiber is studied by using a thermal gravimetric analysis (TGA). Typically beyond 170°C, the wood-fiber devolatilization rate increases. Therefore, it is logical to assume that the degradation of wood-fiber is also severer

15   beyond 170°C. This leads to two important conclusions. First, the drying temperature of wood-fibers is preferably maintained below 170°C, no matter which method of drying is adopted, such as on-line devolatilizing through a vent, oven drying, hot air convective drying, drying in K-mixer and the like. It should be noted that on-line devolatilization can also occur through the partially filled feed-section of the first extruder, in which

20   case the vent may not be needed at all. Second, the maximum temperature in the processing is preferably maintained below 170°C.

Although preference is given to a drying temperature lower than 170°C in order

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to prevent any damage to wood-fibers from thermal degradation, a short time exposure

of wood-fibers to a temperature higher than 170°C, for drying or devolatilization, has

proved to produce a fine-celled foam structure, without significant degradation of

mechanical properties or any undesirable composite structures. Acceptable results

5   have been obtained with the wood-fiber drying or devolatilizing temperature as high as

185°C. This holds true for all the processes (A to F) described in a later section.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention will now be described by way of example only, with

10   reference to the accompanying drawings, in which:

Fig. 1a is a schematic diagram of an extrusion process of the present

invention and showing the injection of a physical blowing agent;

Fig. 1b is a schematic diagram of an extrusion process similar to that

shown in figure 1a but showing a cooling extruder;

15   Fig. 1c is a schematic diagram of an extrusion process similar to that

shown in figure 1a but showing a single screw extruder instead of a twin screw

extruder;

Fig. 1d is a schematic diagram of an extrusion process similar to that

shown in figure 1a but showing a single screw extruder instead of a twin screw extruder

20   and a cooling extruder;

Fig. 2a is a schematic diagram of an alternate extrusion process of the

present invention and showing the injection of a physical blowing agent and the use of

a cascade devolatilisation system having a twin screw extruder followed by a single screw extruder;

      Fig. 2b is a schematic diagram of an extrusion process similar to that shown in figure 2a but showing a cooling extruder;

5   Fig. 2c is a schematic diagram of an extrusion process similar to that shown in figure 2a but showing a twin screw extruder instead of a single screw extruder as the second extruder;

      Fig. 2d is a schematic diagram of an extrusion process similar to that shown in figure 2a but showing a twin screw extruder instead of a single screw extruder

10   as the second extruder and a cooling extruder;

      Fig. 3a is a schematic diagram of an extrusion process of the present invention and using a chemical blowing agent;

      Fig. 3b is a schematic diagram of an extrusion process similar to that shown in figure 3a but showing a cooling extruder;

15   Fig. 3c is a schematic diagram of an extrusion process similar to that shown in figure 3a but showing a single screw extruder instead of a twin screw extruder;

      Fig. 3d is a schematic diagram of an extrusion process similar to that shown in figure 3a but showing a single screw extruder instead of a twin screw extruder

20   and a cooling extruder;

      Fig. 4a is a schematic diagram of an alternate extrusion process of the present invention using a chemical blowing agent and showing the use of a cascade

devolatilisation system having a twin screw extruder followed by a single screw extruder;

Fig. 4b is a schematic diagram of an extrusion process similar to that shown in figure 4a but showing a cooling extruder;

5   Fig. 4c is a schematic diagram of an extrusion process similar to that shown in figure 4a but showing a twin screw extruder instead of a single screw extruder as the second extruder;

Fig. 4d is a schematic diagram of an extrusion process similar to that shown in figure 4a but showing a twin screw extruder instead of a single screw extruder

10   as the second extruder and a cooling extruder;

Fig. 5 is a temperature/foam density graph comparing changes in density of wood fibre composite foam structures with changing barrel temperature at three different devolatization vent temperatures;

Fig. 6 are scanning electron micrographs of wood fibre composite foam

15   structures at different barrel temperatures;

Fig. 7 is a temperature/foam density graph comparing changes in density of wood fibre composite foam structures with changing die temperature at three different barrel temperatures for a fixed vent temperature;

Fig. 8 is a schematic diagram of an extrusion process similar to that

20   shown in figure 2 a & c but showing a single twin screw extruder with vent holes instead of a vent at the interconnection of two extruders; and

Fig. 9 is a schematic diagram of an extrusion process similar to that

shown in figure 4 a & c but showing a single twin screw extruder with vent holes

instead of a vent at the interconnection of two extruders.

DETAILED DESCRIPTION OF THE INVENTION

5   The objective of the invention is to produce fine-celled plastic/wood-fiber

composite foams. A fine-celled structure is characterized by uniform distribution of a

very large number of cells or voids. In order to ensure fine-celled morphology, the

contents of moisture and other volatiles such as extractives of wood-fibers need to be

reduced as much as practicable, using any of the standard drying techniques, such as,

10   online devolatilization, oven drying, hot air convective drying, drying in K-mixer and the

like. Generally the wood-fibres are dried as much as possible and heated to a

temperature below the degradation temperature. That is the wood fiber should not be

heated so high that the structural integrity of the wood is degraded. In the moisture or

volatile-free mixture, a chemical blowing agent (CBA) or a physical blowing agent

15   (PBA) is introduced with other additives, and the processing parameters are optimized

so that the blowing gases are finely dispersed or dissolved into the polymer matrix

under high temperature and pressure. The molten composite mixture or solution is

subjected to a rapid pressure drop under predetermined processing conditions to

produce fine-celled plastic/wood-fiber foams. Moreover, optionally adopting techniques

20   for reducing the processing temperature can assist the shaping of extrudate and the

control of foam density.

The above-described method can be embodied in at least the following six

processes:

Process A: Drying or devolatilizing the moisture and other volatiles such as

extractives, using any conventional technique, at a temperature lower than the

degradation temperature and generally lower than 170°C or slightly higher and using

5   the PBA foaming process, in which the maximum processing temperature is

maintained lower than the active volatilization temperature, generally 170°C, to

generate a fine-celled structure in the plastic/wood-fiber composites.

Process B: Devolatilizing the moisture and other volatiles, through a vent at the

intersection of two cascading extruders, at a temperature lower than the degradation

10   temperature and generally lower than 170°C and using the PBA foaming process, in

which the maximum processing temperature is maintained lower than active

volatilization temperature, generally 170°C, to generate a fine-celled structure in the

plastic/wood-fiber composites.

Process C: Drying or devolatilizing the moisture and other volatiles, using any

15   conventional technique, at a temperature lower than the degradation temperature and

generally lower than 170°C or slightly higher and using the CBA foaming process, in

which the maximum processing temperature is restricted to the minimum level required

to decompose the blowing agent and preferably is less than the active volatilization

temperature, to generate a fine-celled structure in the plastic/wood-fiber composites.

20   Process D: Devolatilizing the moisture and other volatiles, through a vent at the

intersection of two cascading extruders, at a temperature lower than the decomposition

temperature of the CBA and preferably lower than the degradation temperature, and

using the CBA foaming process, in which the maximum processing temperature is

restricted to the minimum level required to decompose the blowing agent and

preferably is lower than the active volatilization temperature, to generate a fine-celled

5   structure in the plastic/wood-fiber composites.

Process E: Utilizing the volatile emissions given out by the wood-fiber to generate a

fine-celled structure in the plastic/wood-fiber composites without using any blowing

agent, and using a single extrusion system with pre-dried wood-fibers, in which

substantial volatile material has been removed during the drying process.

10   Process F: Utilizing the volatile emissions given out by the wood-fiber to generate a

fine-celled structure in the plastic/wood-fiber composites without using any blowing

agent, and using a tandem extrusion system with a devolatilizing vent using un-dried

wood-fibers.

Process A

15   For process A, the present invention focuses on removal of moisture and/or

other volatiles such as extractives of wood-fiber and utilization of a gaseous PBA for

fine-celled foaming of plastic/wood-fiber composite foams with a significantly improved

cell structure. The main advantages of using an inert PBA, are that it is non-hazardous

and more economical than other blowing agents. The PBA used can be any of the

20   non-reactive gases such as CO_2, N_2, He, Ar, Air, etc., or a mixture of these gases in

any combination and proportion. When the PBA is injected in the extruder barrel, it

usually acquires a supercritical state. The processing temperatures and pressures in the barrel are usually sufficiently high for this to happen, even though the injected PBA may be in a liquid, gas or super critical state. The supercritical point, i.e. the critical temperature and the critical pressure, of most of the non-reactive gases is lower than

5   the conditions prevailing in the extruder barrel, so that the above statements apply to nearly all of them.

The processing conditions in the barrel are maintained so as to dissolve, or well disperse, all the gas in the supercritical state into the polymer matrix. The dissolution of the gas into the polymer will depend upon the solubility of the gas in the polymer at

10   the barrel temperature and pressure. We can choose to inject, either a soluble amount of gas or, if needed, an amount greater than the solubility limit and cause it to be finely dispersed by adjusting the conditions in the barrel. Since the solubility of gas in the polymer is almost proportional to the pressure, the injected gas will not be completely dissolved in the polymer if the pressure is lower than the solubility pressure. Although

15   the cell structure is observed to be better, in general, by completely dissolving the gas, according to our experimental results, very fine cell structures have also been achieved by finely dispersing the gas bubbles using an effective mixing device. Whether the injected gas completely dissolves in the polymer or not, a fine cell foam can be made from the plastic/wood-fiber composites using the developed systems as long as the

20   volatiles are not generated from the wood-fibers during processing.

Extensive experimental results show that the volatiles, including moisture,

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coming out of the wood-fiber during extrusion processing in general deteriorate the cellular structure of plastic/wood-fiber composite foams. In order to produce a fine-celled structure, the role of the blowing agent should be dominant and the role of the volatiles should be minimized. In order to minimize the amount of volatiles coming out

5   of wood-fibers during processing, the wood-fiber should be dried or devolatilized enough and the processing temperature should be maintained low enough so that additional evaporation of volatiles is reduced.

In brief, the strategy adopted is that, the wood-fibers are pre-dried or pre-devolatilized by using any one of the commonly used industrial methods, such as, oven

10 drying, hot air convective drying, drying in K-mixer and the like, while maintaining the drying temperature below the degradation temperature and generally lower or slightly higher than 170°C. Then the dried wood-fiber is mixed with the polymer powder/pellets, a coupling agent, and a cell-nucleating agent, such as talc, in a dry blending tumbler to generally achieve homogeneity. The blended mixture is then fed into an extruder

15 through a feeder. A special feeder may be required because the natural adhesiveness of the wood fiber may prevent the mixture from being gravity-fed into the extruder from the hopper. The pre-dried wood-fibers and additives are plasticized and uniformly dispersed in the plastic matrix, in the initial section of an extruder. The PBA is injected and dispersed or dissolved in mid and final zone of the extruder. The temperature of

20 the melt can be reduced optionally and finally it is extruded through a die with a rapid pressure drop (more than 1000 psi across the die) to induce large nucleation. The

temperature of the melt may not have to be decreased for high-density applications. Controlling the die and melt temperatures controls the expansion ratio.

In order to maximize the removal of the volatiles, including moisture, while preventing degradation, the wood-fibers are dried at a temperature below the

5   degradation temperature and generally lower or slightly higher than 170°C. In extrusion processing, care should be taken that the processing temperature does not exceed the drying temperature. It was found that at higher temperatures, the moisture is continually released at a gradual rate, even if it has been completely dried at that temperature. In other words, additional moisture is released even though the wood-

10   fiber has been pre-dried at the processing temperature, and the amount of this moisture will depend upon the extrudates' residence time and processing temperature in the equipment. Thus, an additional important processing parameter is to minimize the residence time so that the moisture content can be minimized while maintaining or preferably decreasing the processing temperature below the active volatilization

15   temperature generally below 170°C.

The use of PBA is particularly suited for this process, as the higher temperature required to decompose a CBA is not needed. Another beneficial effect of using the inert gas CO_2 or N_2 (PBA) is that it has a plasticizing effect on the polymer/wood-fiber mixtures, therefore, not only would it aide in the dispersion of wood-fiber in the plastic,

20   but would also enable higher content of wood-fiber to be used. The plasticizing effect may also enable materials, such as polypropylene (PP) and polystyrene (PS) for which

a higher processing temperature is needed, to be processed at temperatures lower

than 170°C, so that the detrimental effect of moisture or other volatiles may be

reduced.

Process Description: Figures 1 (a-d) show the different configurations possible

5   for this process. In the preparatory stage, the wood-fibers are pre-dried or devolatilized

by using any one of the commonly used industrial methods, such as, oven drying, hot

air convective drying, drying in K-mixer and the like, while keeping the drying

temperature below or slightly above 170°C. This is necessary to ensure that the

volatiles do not evolve and deteriorate the cell morphology of the final extrudate in

10   extrusion processing. Then the dried wood-fibers are mixed with the polymer

powder/pellets, a coupling agent, and a nucleating agent, such as talc, in a dry

blending tumbler. The blended mixture is then fed into an extruder 20 through a feeder

22. In this example the extruder 20 is a twin screw extruder but it could also be a single

screw extruder.   A special feeder may be used because the natural adhesiveness of

15   the wood fiber may inhibit the mixture from being gravity-fed into the extruder 20 from

the hopper.

This mixture is plasticated and further mixed in the initial sections of the screw.

In the mid zone of the extruder, a metered amount of physical blowing agent (PBA) is

injected into the polymer/wood fiber matrix at 24. For example as shown in Figure 1a,

20   the PBA is CO_2 which is stored in a PBA tank 26. A positive-displacement pump 28

may be used to inject the PBA into the mixture. A numeric controller or valve 30 is

- 18 -

used to meter the flow of the PBA into the mixture. A second valve 31 is used for filling

the pump with PBA from the PBA tank. During this filling process, the numeric

controller or valve 30 between the pump 28 and the extruder 26 is closed. During

extrusion foaming operation, the second valve 31 is closed and numeric controller is

5   opened. This mixture is then subjected to high shear forces by the screw and/or by

any type of mixers, in the presence of high pressure (typically the pressure is higher

than 2000 psi). This ensures the complete dissolution or thoroughly uniform dispersion

of the PBA gases into the polymer matrix. During this process some additional

moisture or volatiles may be released from the wood-fiber, but this is difficult to avoid.

10   To reduce these emissions, the residence time in extruder 20 should be minimized.

Optimally, the melt temperature of the polymer/gas mixture can be homogeneously

reduced in a heat exchanger 32. A commonly well-known static mixer in a cooling heat

exchanger wherein the mixer has vanes positioned such that the core fluid is

transported away from the center and the peripheral material is moved to the core. The

15   mixer housing is enclosed in a jacket through which the cooling fluid flows. Such a

mixer is shown in Figure 1a may be used. The mixture passes from the heat

exchanger 32 to a die 36.

For high wood-fiber content a cooling extruder 34, shown in Figure 1b, may be

used as a cooling device instead of a heat exchanger with a static mixer. Added cost

20   of an extruder can be justified by achieving higher pressure at the die if cooling is

required. The extrudate finally passes through the die where its pressure is rapidly

dropped, which produces a large number of nucleation sites due to the Spinodal

decomposition of polymer and gas. The gas from the adjacent polymer matrix diffuses

into these sites and causes expansion. The temperature of the die can be controlled so

as to freeze the surface layer of the polymer, which prevents the diffusion of gases into

5   the atmosphere. This in turn helps in achieving expansion. The die temperature can

also be lowered to control the unfoamed skin thickness of the extrudate for the profile

dies. The profile dies can be based on the Celuka die [23, 24], where a dividing

mandrel is inserted and foaming is induced to occur inward, The art of Celuka die is

well known as described in references 23 and 24. The profile die can also be of other

10   non-Celuka designs.

Figures 1c and 1d illustrate the use of a single screw extruder 38 instead of a

twin screw extruder shown in figures 1a and 1b. Otherwise the configurations shown in

Figure 1c and 1d correspond with Figures 1a and 1b, respectively.

Example for Process A

15   Material

High-density polyethylene (HDPE 2710) supplied by Nova Chemicals was

utilized in the experiments. The 50wt% wood-fibers used were standard softwood

(pine) grade 12020, supplied by American Wood Fibers. The physical blowing agent

(PBA) was commercial CO_2 from Matheson Gas Products. 3wt% coupling agent was

20   used for improving the adhesion between the hydrophobic PE and the hydrophyllic

wood-fiber. The coupling agent was Fusabond adhesive resin E-MB-100D from

Dupont, Canada. Wood-fibers were dried in vacuum oven at 105C° for 12 hours to

remove the moisture and volatiles. All the materials were used as received

Experimental setup and procedure

A schematic of the equipment for process A is shown in Figure 1a. In the

5   preparatory stage, wood-fiber oven-dried at 105°C for 12 hours is mixed with the

polymer powder/pellets, a coupling agent, and a nucleating agent such as talc in a dry

blending tumbler, and fed into the extruder 20, using a feeder 22.

A metered amount of CO_2 (4 wt%) was injected into the molten polymer/wood-

fiber mixture at 24 in the barrel, using a positive displacement pump. In experimental

10   setup herein the injection site was about $2/3^{rd}$ away from inlet. However, it will be

appreciated by those skilled in the art that the actual location is not important as long

as the polymer has been already melted completely by the time the melt gets to the

location of the gas-injection port and as long as the pressure at this port is high enough

to be able to dissolve all the injected gas. The pressure in the extruder and

15   downstream was maintained high enough to expedite dissolution of CO_2 into the plastic

matrix. However, the processing temperature was not increased over 155°C to

minimize the release of further moisture or volatiles and to prevent the thermal

degradation of wood-fiber. The temperature of the melt was then homogeneously

lowered in a heat exchanger 32. Finally, it was extruded out through a die 36 where the

20   surface temperature was further reduced to stabilize the foam. Samples were taken at

various nozzle temperatures for density measurement and cell morphology

- 21 -

characterization

Results

The removal of moisture and other volatiles, injection of CO_2, dissolution of CO_2, cooling, and the rapid pressure drop (more than 1000 psi across the die) resulted in a

5   significant improvement of the cell structure of foams. The scanning electron micrographs (SEMs) of the foams produced on the system described herein clearly reveal that the cell structure was much more regular and smaller in size when compared to the foam structure obtained using un-dried wood-fibers. The largest cells were smaller than 200 microns and the distribution of cell size was very uniform. The

10   density of HDPE/wood-fiber composite foams was successfully controlled to be in the desired range of 0.5 g/cm^3 to 0.8 g/cm^3 by controlling the amount of PBA and the die temperature.

The surface quality was dramatically improved with the dried wood-fibers. Foams obtained from un-dried wood-fibers from single extruder system exhibited a

15   very non-uniform cell structure and a rough surface. The cross-sectional dimensions were very irregular and blister like bubbles were present on the surface. For the foams produced by the process described herein, the surface finish was smooth and regular.

Process B

20   Most of the prior art drying methods for drying the wood fibers suffer from two disadvantages. Firstly, they involve costly batch processing which is not desirable for

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continuous processes. Secondly, the wood-fiber cannot be heated to a temperature

higher than 100°C for a longer period without causing its degradation with the presence

of Oxygen. To overcome some of these disadvantages cascade extruders may be

used for devolatilization and removal of moisture and other volatiles while the molten

5   plastic is transferred from one extruder (or compounder) to the second extruder in a

tandem or cascade arrangement. Controlling the material feed rate from the first

extruder into the second extruder controls the material loss at the vent. If there is any

flow out of the vent, it can be suppressed by reducing the rotational speed of the first

extruder and/or by reducing the feed rate into the hopper.

10

Process Description: Process B is a subset of process A in which online

devolatilization, instead of separate batch process drying, is used for purging out the

moisture and other volatiles from the plastic/wood-fiber/additives mixture. The strategy

adopted is that, the un-dried wood-fibers, plastic and additives are dry blended and the

15   blended mixture is then fed into an extruder through a feeder 22. The first extruder 40

can be a single or twin-screw mixing extruder or compounder, where plastication and

mixing takes place. During this process most of the moisture and other volatiles are

evaporated due to the elevated temperature (typically between 145°c to 170°C). The

molten mixture is then devolatilized at a vent 41 in a cascade extrusion system and

20   conveyed into the second extruder. On-line devolatilization can be accomplished within

the first extruder if a twin screw extruder is used as the first extruder, and devolatilizing

ports 51 can be opened along the screws as shown in Figures 8 and 9.

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In the second extruder 42, a metered amount of physical blowing agent (PBA) is

injected into the polymer/wood fiber matrix at 44. This mixture is then subjected to high

shear forces by the screw and/or by any type of mixers, in the presence of high

pressures (typically the pressure is above 2000 psi). This ensures the complete

5   dissolution or thoroughly uniform dispersion of the PBA gases into the polymer matrix.

During this process some additional moisture or volatiles may be released from the

wood-fiber, but this is difficult to avoid. To reduce these emissions, the residence time

should be minimized. Optionally, the melt temperature of the polymer/gas mixture can

be homogeneously reduced in a heat exchanger 32. A commonly well-known static

10   mixer in a cooling heat exchanger shown in Figure 2a can be used.

As above, for high wood-fiber content a cooling extruder 34, shown in Figure 2b,

can be used as a cooling device instead of a heat exchanger 32 with a static mixer.

The extrudate finally passes through the die 36 where its pressure is rapidly dropped,

which produces a large number of nucleation sites due to the Spinodal decomposition

15   of polymer and gas. The gas from the adjacent polymer matrix diffuses into these sites

and causes expansion. The temperature of the die can be controlled by a cooling

jacket and a band heater so as to freeze the surface layer of the polymer, which

prevents the diffusion of gases into the atmosphere. This in turn helps in achieving

expansion. The die temperature can also be lowered (from around 150°C down to

20   melting temperature of PE, which is typically around 130°C) to control the unfoamed

skin thickness of the extrudate for the profile dies. The profile dies can be based on the

Celuka die [23, 24], where a dividing mandrel is inserted and foaming is induced to occur inward. The profile die can also be of other non-Celuka designs.

In regard to processes A and B, the results were observed to be best when the highest processing temperature was maintained lower than 170°C. It seems that

5   processing the composites under 170°C substantially prevented the generation of moisture and other volatiles. The TGA analysis also confirmed this observation, as it was observed that the weight loss rate of wood-fibers increased at temperatures higher than 170°C.

It seemed that a severe degradation of wood-fiber occurs and thereby severe

10   deterioration of mechanical properties of plastic/wood-fiber composites occurs above 205°C. However, even before the degradation of the mechanical properties of wood-fiber, there seems to be an excessive amount of weight loss above 170°C, according to the TGA results. This is probably due to the evaporation of some of the extractives/volatiles, when heated above 170°C. Although the mechanical properties

15   may not be appreciably affected due to loss of some of these extractives/volatiles, the evaporation of these materials significantly contributes to the deterioration of foam structure. Therefore, it is preferably to reduce these emissions by minimizing the processing temperatures and residence time, even if the wood-fibers were well devolatilized before processing.

20   **Example for Process B**

Material

HDPE Grade 2710 from Nova was used as the base polymer for the

composites. 50 wt% standard wood-fibers softwood grade 12020, supplied by

American Wood Fibers, were used. 3 wt% coupling agent (Fusabond adhesive resin E-

MB-100D from Dupont, Canada) was used for improving the adhesion between the PE

5   and the wood-fiber. Commercial CO_2, upto 4wt%, from Matheson Gas was used as the

PBA. All the materials were used as received.

Experimental setup and procedure

A schematic of the equipment for process B is shown in Figure 2a. In the

preparatory stage, un-dried wood-fiber is mixed with the polymer powder/pellets, a

10   coupling agent, and a nucleating agent such as talc in a dry blending tumbler, and fed

into the first twin-screw extruder 40, using a feeder 22. The temperature at the exit of

this extruder was maintained at the highest processing temperature, which in this case

was 180°C, for completely vaporizing the moisture and other volatiles from the wood-

fiber. The output of this extruder was fed into the second extruder. At the

15   interconnection, which was open to atmosphere, generally all the vaporized volatiles

were devolatilized and the moisture and generally volatile-free mixture was conveyed

into the second extruder by the motion of the screw.

A metered amount of CO_2 was injected into the molten polymer/wood-fiber

mixture at 44 in the middle of the barrel of the second extruder 42, using a positive

20   displacement pump 28. The pressure in the second tandem extruder 42 and

downstream was maintained high enough (typically the pressure is above 2000 psi) to

expedite dissolution of CO_2 into the plastic matrix. The temperature of the melt was then homogeneously lowered in a heat exchanger 32. Finally, it was extruded out through a die 36 where the surface temperature was further reduced to stabilize the foam. Samples were taken at various nozzle temperatures for density measurement

5   and cell morphology characterization.

Results and discussion

The foam density of HDPE/wood-fiber composites produced, using the tandem extrusion system with a devolatilizing vent at the interconnection was successfully controlled in the range of 0.5 to 0.8 g/cm^3. The cell morphology of the foams produced

10   was very uniform with the largest cell size being under 150 microns. The surface quality of the foams was very good too.

Process C

For process C, the present invention focuses on moisture removal and utilization of a CBA for fine-celled foaming of plastic/wood-fiber composite foams with a

15   significantly improved cell structures. The basic strategy adopted is same as described for process A, with the difference that the CBA is dry blended along with plastic and wood-fiber before being fed into an extruder in which the resin is plasticated and the mixture is conveyed further into the extruder where it is heated so that the CBA is decomposed and releases all its gaseous contents. Further processing is again similar

20   to that described for process A.

Process Description: In the preparatory stage, the wood-fibers are pre-dried by

using any one of the commonly used industrial methods, such as, oven drying, hot air

convective drying, drying in K-mixer and the like, while maintaining the drying

temperature below or slightly higher than 170°C. The drying temperature can be even

very high up to the decomposition temperature of the CBA to be used, which may

5   cause slight degradation of the wood-fiber. However. drying over 205°C should be

avoided because of the severe degradation of the wood-fiber, This reduces the

moisture and other volatiles, and thereby reduces the deterioration of the cell

morphology of the final extrudate. Then the dried wood-fiber is mixed with the polymer

powder/pellets, a CBA, a coupling agent, and a nucleating agent, such as talc, in a dry

10   blending tumbler. The blended mixture is then fed into an extruder 20 through a feeder

22. This arrangement is shown in figure 3a and is similar to those discussed above in

regard to Figures 1a and 2a but there is no injection of a PBA.

  This mixture is plasticated and further mixed in the initial sections of the screw.

In the last zone 23 of the extruder the temperature is increased so as to decompose

15   the CBA. During this process some additional moisture or volatiles may be released

from the wood-fiber, but this is difficult to avoid for CBA processing unless the drying

temperature was as high as the decomposition temperature of CBA. To reduce these

emissions, the residence time should be minimized. This mixture is then subjected to

high shear forces by the screw and/or by any type of mixers, in the presence of high

20   pressures (typically the pressure is above 2000 psi). This facilitates the dissolution or

dispersion of the decomposed gases into the polymer matrix. The melt temperature of

the polymer/gas solution is reduced to about 150°C. Lower limit is determined by

material and its MFI. In some cases it can go even below 140°C in a heat exchanger
22. A commonly well-known static mixer in a cooling heat exchanger shown in Figure
3a can be used.

For a high wood-fiber content, a cooling extruder 34, as shown in Figure 3b, can

5   be used as a cooling device instead of a heat exchanger with static mixer. The
extrudate finally passes through the die where its pressure is rapidly dropped, which
produces a large number of nucleation sites due to the Spinodal decomposition of
polymer and gas. The gas from the adjacent polymer matrix diffuses into these sites
and causes expansion. The temperature of the die can be controlled so as to freeze

10   the surface layer of the polymer, which prevents the.diffusion of gases into the
atmosphere. This in turn helps in achieving expansion.

Figures 3c and 3d show the other configurations possible for this process.
Specifically Figure 3c is similar to Figure 3a but shows a single screw extruder 38
rather than a twin screw extruder 20. Similarly Figure 3d is similar to Figure 3b and it

15   shows a single screw extruder 38 rather than a twin screw extruder 20.

Process D

As discussed above, an efficient method, which offers many advantages, is to
use cascade devolatilization and to remove moisture while the molten plastic is
transferred from one extruder (or compounder) to the second extruder in a tandem

20   arrangement.   This may also be used in association with the CBA process.

Process Description: Process D is a subset of process C in which online

devolatilization is used for substantially purging out the moisture and other volatiles

from the plastic/wood-fiber/additives mixture. In the preparatory stage, the un-dried

wood-fiber is mixed with the polymer powder/pellets, a CBA, a coupling agent, and a

nucleating agent, such as talc, in a dry blending tumbler. The blended mixture is then

5   fed into the first extruder 20 through a feeder 22.   This arrangement is similar to that

shown in figure 2a but it does not include the injection of a PBA.

The first extruder 40 can be a single or twin-screw mixing extruder or

compounder, where plastication and mixing takes place. During this process most of

the moisture and other volatiles are evaporated through the hopper due to the elevated

10   temperature. The molten mixture is then devolatilized at a vent in a cascade extrusion

system and conveyed into the second extruder 42. Care should be taken that the

temperature at the vent should not be high enough to decompose the CBA. As noted

previously, on-line devolatilization can be accomplished within the first extruder if a twin

screw extruder is used as the first extruder, and devolatilizing ports can be opened

15   along the screws.

In the second extruder 42, the temperature of the molten mixture is elevated so

as to decompose the CBA. During this process some additional moisture or volatiles

will be released from the wood-fiber, but this is difficult to avoid. To reduce these

emissions, the residence time should be minimized. This mixture is then subjected to

20   high shear forces by the screw and/or by any type of mixers, in the presence of high

pressures and temperatures. This facilitates the dissolution or dispersion of the gases

evolved from the CBA, into the polymer matrix. The melt temperature of the polymer/gas mixture is reduced in a heat exchanger. A commonly well-known static mixer in a cooling heat exchanger shown in Figure 4a can be used.

For high wood-fiber content a cooling extruder 34, as shown in Figure 4b, can

5   be used as a cooling device instead of a heat exchanger with static mixer. The extrudate finally passes through the die where its pressure is rapidly dropped, which produces a large number of nucleation sites due to the Spinodal decomposition of polymer and gas. The gas from the adjacent polymer matrix diffuses into these sites and causes expansion. The temperature of the die can be controlled so as to freeze

10   the surface layer of the polymer, which prevents the diffusion of gases into the atmosphere.

Example for Process D

Material

HDPE Grade 58G from Novacor was used as the base polymers for the

15   composites. The wood-fibers were standard softwood (pine) grade 12020, supplied by American Wood Fibers. The CBA, Hydrocerol Compound (HC), was supplied by Boehringer Ingelheim. The coupling agent used for improving the adhesion between the PE and the wood-fiber was Fusabond adhesive resin E-MB-100D from Dupont, Canada. All the materials were used as received.

20   Experimental setup and procedure

The cascade or tandem extrusion system shown in Figure 4a was used in the

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experiments. The mixtures were dry blended and fed into the first twin-screw extruder

40, using a feeder 22. The temperature in this extruder was maintained at a level

sufficient for vaporizing the moisture (145°C to 170°C) **GIVE A RANGE**, but not so high

as to cause the decomposition of CBA and the degradation of wood-fiber. The output

5   of this extruder was fed into the second extruder 42. At the interconnection, which was

open to atmosphere, substantially all the vaporized water and other volatiles were

devolatilized and the moisture and volatile-free mixture was conveyed into the second

extruder 42 by the motion of the screw. The temperatures in the second tandem

extruder 42 were maintained at 155°C and 165°C in zones 1 (43) and 2 (45),

10   respectively. The CBA was decomposed in zone 3 (47) and the released gases were

dissolved in the polymer matrix under the action of high shear at elevated temperature

and pressure. The temperature of the melt was then lowered (typically to150°C) in a

heat exchanger 22. Finally, it was extruded out through the die 36 where the surface

temperature was further reduced (to about 150°C. Lower limit is determined by material

15   and its MFI. In some cases it can go even below 140°C) to increase the stabilization of

the foaming process. Samples were taken at various die temperatures for density

measurement and cell morphology characterization.

Results and discussion

Using this system, composite foams could be produced with controllable foam

20   density varying from 0.5 g/cm^3 to 0.8 g/cm^3. These composites also exhibited uniform

cell distribution and superior surface quality as compared to those produced on a non-

devolatilized system. The generation of large sized cells was successfully suppressed, and the cell population density was increased by one order of magnitude. Therefore, the uniformity of cell structure was significantly improved by adopting the process described herein.

5   The use of CBA requires some special material and processing considerations. The processing temperature in the first extruder and the vent should be lower than the decomposition temperature of the blowing agent. The onset of decomposition for HC, the blowing agent used, is 160°C, so the previously mentioned temperatures should not exceed this limit. This would suggest that the polyethylene materials could be

10   effectively foamed with a HC on this system.

Process E

For process E, the present invention uses the volatile emissions from the wood-fibers for generating a fine-celled foamed structure without utilizing any other blowing

15   agent. The wood-fiber will release moisture or volatiles at high temperatures as mentioned earlier. These volatiles, in general, will affect foaming negatively, especially at a larger content. But with certain materials and under certain processing conditions, as long as the amount of volatiles is not high, these volatiles can be utilized to generate an acceptable cell structure in plastic/wood-fiber composite foams having

20   good surface properties as well. Usually, the higher the temperature, the greater the amount of moisture/volatiles released. In order to suppress the adverse effects of the

excessive moisture/volatiles on the cell structure, we can lower the processing

temperature is lowered to a reasonable degree and the foaming process is controlled

while utilizing these emissions.

5   **Process Description:**   Process E utilizes same equipment as process C in which

no other blowing agent is used. The material is pre-dried by any of the conventional

methods, such as oven drying, K-mixer blending or hot air-drying so that most of the

moisture and other volatiles are removed either from the wood-fibers or from the

plastic/wood-fiber/additives mixture. If dried wood-fibers are used, they are mixed with

10   plastic and coupling agent and dry blended. If a K-mixer is used, the mixture is

granulated in a granulator. The blended mixture is then fed into the extruder 20

through a feeder 22 if dried wood-fibers are used or directly into the hopper if K-mixer

was used.   The equipment used is same as in Figure 3a and the processing steps are

also same as in process C, except the heating temperature is not determined by the

15   decomposition temperature of chemical blowing agent.

Care should be taken that the barrel temperature should not be too high to

generate excessive volatiles (below 170°C). The temperature of the molten mixture is

controlled so that a limited amount of moisture or volatiles can be released from the

wood-fiber, which is used for foaming. Consider a specific case of cascade extrusion

20   processing at 170°C, in which the residence time in the first extruder is 3 minutes and

the processing time in the second extruder is 2 minutes. In addition to loosing all the

moisture, the wood fibers will loose an additional 0.65% weight in the first extruder.

These wood fibers will loose a further 0.2% weight while being processed in the

second extruder, part of which will be entrapped and contribute to foaming. The

amount of volatile emissions which contribute to foaming can be controlled by varying

5   the processing temperature and the residence time in the extruder. This mixture is

then subjected to high shear forces by the screw and/or by any type of mixers, in the

presence of high pressures and temperatures. Temperatures and pressures are

measured and the temp. can be controlled by band heaters and the pressure by

controlling the flow rate. This facilitates the dissolution or dispersion of the gases

10   emitted by the wood-fiber, into the polymer matrix. The melt temperature of the

polymer/gas mixture is reduced (typically to 150°C) in a heat exchanger. A commonly

well-known static mixer in a cooling heat exchanger shown in Figure 3a can be used.

For high wood-fiber content a cooling extruder 34, as shown in Figure 3b, can

be used as a cooling device instead of a heat exchanger with static mixer. The

15   extrudate finally passes through the die where its pressure is rapidly dropped, (more

than 1000psi across the die) which produces a large number of nucleation sites due to

the Spinodal decomposition of polymer and volatiles. The volatiles from the adjacent

polymer matrix diffuses into these sites and causes expansion. The temperature of the

die can be controlled so as to freeze the surface layer of the polymer and/or to

20   decrease the diffusivity of volatiles, which prevents the diffusion of gases into the

atmosphere.

Process F

For process F, the present invention envisages the use of the volatile emissions from the wood-fibers for generating a fine-celled foamed structure without utilizing any other blowing agent. The concept of using these emissions from the wood-fiber is same as described for process E, the only difference being that the excessive moisture

5   and volatiles are removed through a vent in a tandem system.

Process Description: Process F utilizes same equipment as of process D while no other blowing agent is used. Online devolatilization is used for purging out most of the moisture and other volatiles from the plastic/wood-fiber/additives mixture. In the preparatory stage, the un-dried wood-fiber is mixed with the polymer powder/pellets

10   and a coupling agent, in a dry blending tumbler. The blended mixture is then fed into the first extruder 20 through a feeder 22.   The equipment used is same as in Figure 4a and the processing steps are also same as in process D.

The first extruder 40 can be a single or twin-screw mixing extruder or compounder, where plastication and mixing takes place. During this process, most of

15   the moisture and other volatiles are evaporated through the hopper due to the elevated temperature. The molten mixture is then devolatilized at a vent in a cascade extrusion system and conveyed into the second extruder 42. Care should be taken that the temperature at the vent should not be high enough to degrade the wood-fibers. On-line devolatilization can be accomplished within the first extruder if a twin screw extruder is

20   used as the first extruder, and devolatilizing ports can be opened along the screws.

In the second extruder 42, the temperature of the molten mixture is controlled so

that a limited amount of moisture or volatiles can be released from the wood-fiber, which is used for foaming. This mixture is then subjected to high shear forces by the screw and/or by any type of mixers, in the presence of high pressures and temperatures. This facilitates the dissolution or dispersion of the gases emitted by the

5   wood-fiber, into the polymer matrix. The melt temperature of the polymer/gas mixture is reduced in a heat exchanger. A commonly well-known static mixer in a cooling heat exchanger shown In Figure 4a can be used.

For high wood-fiber content, a cooling extruder 34, as shown in Figure 4b, can be used as a cooling device instead of a heat exchanger with static mixer. The

10   extrudate finally passes through the die where its pressure is rapidly dropped, which produces a large number of nucleation sites due to the Spinodal decomposition of polymer and volatiles. The volatiles from the adjacent polymer matrix diffuse into these sites and causes expansion. The temperature of the die can be controlled so as to freeze the surface layer of the polymer and/or to decrease the diffusivity of volatiles,

15   which prevents the diffusion of gases into the atmosphere.

Examples for Process E

Material

HDPE Grade 2710 from Novacor was used as the base polymers for the composites. The wood-fibers were standard softwood (pine) grade 12020, supplied by

20   American Wood Fibers. The coupling agent used for improving the adhesion between the PE and the wood-fiber was Fusabond adhesive resin E-MB-100D from Dupont,

Canada. All the materials were used as received.

Experimental setup and procedure

The tandem extrusion system shown in Figure 4a was used in the experiments.

The mixtures were dry blended and fed into the first twin-screw extruder 40, using a

5   feeder 22. The temperature in this extruder was maintained at a level sufficient for

adequate processing of material, but not so high as to cause the degradation of wood-

fiber (from 145°C to 170°C). The output of this extruder was fed into the second

extruder. At the interconnection, which was open to atmosphere, all the vaporized

water and other volatiles were devolatilized and the moisture and volatile free mixture

10   was conveyed into the second extruder by the motion of the screw. The temperatures

(from 145°C to 160°C) in the second tandem extruder were maintained at low levels in

zones 1 and 2. Additional gases were evolved from wood-fiber by elevating the mixture

temperature in zone 3 and the released gases were dissolved in the polymer matrix

under the action of high shear at elevated temperature and pressure. Typically the

15   pressure was between 2000 – 3000 psi. A range of temperatures were tested and it

was determined that temperatures between 150°C and 185°C worked.   However, the

barrel temperature should be typically below 170°C. The temperature of the melt was

then lowered in a heat exchanger 22 (to about 150oC) . Finally, it was extruded out

through the die 36 where the surface temperature was further reduced to increase the

20   stabilization of the foaming process.

Two sets of procedures were adopted to produce HDPE/wood-fiber composite

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foams. In the first set of experiments, the devolatilizing vent temperature was set at

three different values, and the highest barrel temperature was varied at 5°C intervals

for each vent temperature. In the second set of experiments, the devolatilizing vent

temperature was kept constant, but the highest barrel temperature was set at three

5   different values, and the die temperature was varied at 5°C intervals for each barrel

temperature.

Results and discussion

Experiments with Varying Barrel Temperature

A set of experiments of HDPE mixed with un-dried wood-fiber and 3% coupling

10   agent, without a blowing agent were carried out to identify the relationship between

processing temperatures and the cell structure, while fixing the drying (i.e.,

devolatilizing) conditions. This was done to find the lowest processing temperature at

which the moisture/volatiles generation from wood-fiber is minimized and the small

amount generated could be utilized for foaming.

15   In this set of experiments, three different devolatilizing temperatures at the vent

were used and for each of these, the barrel temperature in the second extruder was

varied to identify its effect on the cell structure. The die temperature was maintained to

be 140°C.

Figure 5 shows that the foam density of wood-fiber composites was decreased

20   as the barrel temperature in the second extruder increased. At a higher barrel

temperature, the wood-fiber released more moisture/volatiles out, and therefore, the

foaming effects of these volatiles were increased and the foam density was reduced.

For the barrel temperature range of 145°C-170°C, the foam density was around

1.1g/cm^3. Above 170°C, the foam density decreases quickly to 0.85 g/cm^3. This implies

that a lot of moisture/volatiles were released from wood-fiber above 170°C and

5   resulted in significant foaming. Figure 6 shows some typical cell morphologies of the

wood-fiber/HDPE composite foam samples blown without any blowing agents. At the

same devolatilizing temperature of 160°C, when the barrel temperature increased from

145°C, 165°C, to 175°C, it is observed that foaming occurred due to the

volatiles/moisture generated from the wood-fiber, and the corresponding foam density

10   was 1.1 g/cm^3, 1.0 g/cm^3, and 0.87 g/cm^3, respectively. At the barrel temperature

145°C, there was almost no foaming in the composite. As the barrel temperature was

increased to 165°C, some small sized bubbles and voids appeared. When the barrel

temperature was further increased to 175°C, it is obvious that more volatiles were

released from wood-fiber resulting in significant foaming with an irregular cellular

15   structure. This implies that when the amount of generated moisture and volatiles is

significant, due to the high processing temperature, the formation of big voids is difficult

to avoid. Therefore, to minimize the effects of moisture/volatiles on foaming, the

maximum processing temperature in the second extruder should be low, preferably

below 170°C.

20   Figure 5 also shows that the foam density of composites generally increased as

the devolatilizing temperature T_v was increased. This implies the devolatilizing vent is

effective in removing moisture/volatiles from wood-fiber. Another interesting

- 40 -

observation from this figure is that the foam density decreased slightly as the second

barrel temperature T_b increased while $T_b < T_v$. When $T_b > T_v$, the foam density

decreased sharply when the barrel temperature was increased. This shows wood-fiber

will further release moisture/volatiles even after the devolatilization at the vent,

5   because of the higher processing temperature than the drying (i.e., devolatilization)

temperature. Therefore, it is essential to set the barrel temperature lower than the

devolatilizing temperature, to minimize the gaseous emissions additionally generated

during processing, and thereby, to produce a fine celled structure. This inference is

also equally applicable to the pre-drying case of process A and process C.

10

Experiments with Varying Die Temperature

Another set of experiments of HDPE mixed with un-dried wood-fiber and 3%

coupling agent, without a blowing agent were carried out to identify the relationship

between die temperatures and the foam density at three different barrel temperature,

15   while fixing the drying (i.e., devolatilizing) condition at 170°C. This was done to find the

lowest density achievable when the moisture/volatiles generation from wood-fiber is

utilized for foaming, while the amount of moisture and volatile is minimal to produce a

fine-celled structure, by maintaining the second extruder barrel temperature below

170°C. Experiment was also conducted at high second extruder barrel temperature

20   above 170°C for comparison puspose.

Figure 7 shows that the foam density of wood-fiber composites was decreased

significantly as the die temperature decreased, when the barrel temperature was

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175°C. However, because of too high a barrel temperature, the wood-fiber released too much moisture/volatiles, and as a result, irregular-shaped, big-sized cells were observed and the foam structure was very non-uniform. For the barrel temperature of 160°C, the foam density could be lowered below 0.75 g/cm^3, by decreasing the die

5   temperature below 140°C. The foam structure was very fine because of the small amount of volatiles generated at this low barrel temperature. For a barrel temperature of 145°C, no foaming could be obtained. In fact the final density was always more than the density of the individual components indicating that the high extrusion pressures compacted the composite. This also indicates that lower processing temperature

10   suppresses the volatile emission from the wood-fiber which has been dried (i.e., devolatilized) at a highĕr temperature.

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